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Indian Standard

SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

UDC 661·833·532 [661·724] : 665·58

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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May 1986

Indian Standard

SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

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AMENDMENT NO. 3 SEPTEMBER 2012
TO
IS 11487 : 1985 SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

[*Page* 4, *Table* 1, *Sl No.* (v)] — Delete.

[*Page* 4, *Table* 1, *Sl No.* (viii), *col* 3] — Substitute '6.0 to 11.0' *for* '6.0 to 8.0'.

[*Page* 4, *Table* 1, *Sl No.* (xiii), *col* 3, *see also Amendment No*. 2] — Substitute '30' *for* '100'.

(PCD 19)

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(*Continued on page* 19)

Indian Standard

SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 11 December 1985, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Sodium lauryl ether sulphate is chemically $R—O(C_2H_4O)_nSO_3Na$ where R is predominantly C_{12} derived from a coconut or palm kernel source and n is on the average 2 to 2·5. It is primarily used as active detergent in shampoo.

0.3 Commercial cosmetic grades are generally available as 30 percent solution.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test of sodium lauryl ether sulphate (30 percent solution) used in cosmetic industry.

2. REQUIREMENTS

2.1 Description

2.1.1 The material shall be clear, colourless to pale amber, viscous solution.

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*Rules for rounding off numerical values (*revised*).

2.1.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the Table 1.

TABLE 1 REQUIREMENTS FOR SODIUM LAURYL ETHER SULPHATE SOLUTION (30 PERCENT)

SL No.	CHARACTERISTICS	REQUIREMENTS	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Active detergent, percent by mass, *Min*	27	A-2
ii)	Non-detergent organic matter, percent by mass, *Max*	2·5	A-3
iii)	Sodium chloride and sodium sulphate, percent by mass, *Max*	3·5	A-4 and A-5
iv)	Cloud point, °C	Less than 0	A-6
v)	Viscosity, cps at 27°C	1 500 to 5 000	A-7
vi)	Heavy metals (as Pb), parts per million, *Max*	20	A-8
vii)	Arsenic (as As_2O_3), parts per million, *Max*	2	A-9
viii)	*p*H of 5 percent aqueous solution	6·0 to 8·0	A-10
ix)	Iron (as Fe), parts per million, *Max*	20	A-11
x)	Formalin, percent by mass, *Max*	0·04	A-12

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in HDPE carboys or as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and marked with mass of the material in the container; vegetable oil source and molecular mass of the fatty alcohol; manufacturers' name and his trade-mark, if any; batch number in code or otherwise; and the year of manufacture.

3.2.1 The containers shall be marked suitably to indicate whether lauryl alcohol used in the production of the sodium ether lauryl sulphate is synthetic or not.

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standards conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors may be obtained from the Indian Standards Institution.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR SODIUM LAURY ETHER SULPHATE FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF ACTIVE DETERGENT MATTER

A-2.1 Outline of the Method — Sodium lauryl ether sulphate is determined by estimating the combined sulphur trioxide by cationic titration and then calculating the sodium lauryl ether sulphate content from the value of combined sulphur trioxide obtained.

A-2.1.1 *Reagent*

A-2.1.1.1 *Cationic reagent* — Weigh 1·5 ± 0·001 g of cetyl trimethyl ammonium bromide (cetab) in a 250-ml beaker. Add 100 ml of water and stir until dissolved. Transfer quantitatively to a one-litre volumetric flask, mix well and make to volume.

A-2.1.1.2 *Potassium dichromate solution* — 0·01 M; prepared by dissolving 2·942 g of potassium dichromate dried at 120 to 140°C for about 3 hours in water and diluting the solution to 1 litre.

A-2.1.1.3 *Dilute sulphuric acid* — 4 N.

A-2.1.1.4 *Potassium iodide solution* — 10 percent.

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*Specification for water for general laboratory use (*second revision*

A-2.1.1.5 *Sodium thiosulphate solution* — 0·1 N (*see* IS : 2316-1968*).

A-2.1.1.6 *Starch indicator solution* — Triturate 5 g of starch and 0·01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 minutes. Allow to cool and decant off the supernatant clear liquid.

A-2.1.1.7 *Methylene blue indicator solution* — Dissolve 0·1 g of methylene blue in 100 ml of water. Transfer 15 ml of this solution to a one litre volumetric flask and add 50 g of sodium sulphate. Add 500 ml of water and 6·8 ml of concentrated sulphuric acid and shake until solution is complete. Dilute to the mark.

A-2.1.1.8 *Chloroform*

A-2.1.2 *Procedure*

A-2.1.2.1 *Standardization of cationic reagent*

a) Pipette out 50 ml of standard cationic reagent in a beaker and add 25 ml of standard potassium dichromate solution. Heat the beaker to about 90°C to coagulate the precipitate and filter through filter paper (Whatman No. 40 or equivalent). Wash the precipitate thoroughly, collecting the filtrate and washings in a beaker. Add to the beaker 10 ml of dilute sulphuric acid and 10 ml of potassium iodide solution. Titrate the liberated iodine with standard sodium thiosulphate solution, adding starch solution towards the end of the titration.

b) Calculate the normality of cationic reagent by the following formula:

$$N = \frac{0{\cdot}02}{50}\left(25 - \frac{V}{0{\cdot}6}\right)$$

where

N = normality of the cationic reagent; and

V = volume in ml of standard sodium thiosulphate solution used in the titration in **A-2.1.2.1** (a).

A-2.1.2.2 *Titration of the material*

a) Weigh accurately a quantity of the material sufficient to give approximately 0·320 g of combined sulphur trioxide in a 250-ml beaker. (A rough estimate of the quantity required is obtained by dividing 320 by the expected combined sulphur

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*Methods of preparation of standard solution for colorimetric and volumetric analysis (*first revision*).

trioxide content). Use about 700 to 800 ml of warm water to transfer quantitatively to a 1 litre volumetric flask. Warm on a steam bath and shake gently until the material is dissolved and solution is clear. Cool and dilute to the mark with water and mix thoroughly.

b) Pipette out 10·0 ml of the solution from the volumetric flask into a 100-ml glass cylindrical titration vessel. Add into it 25·0 ± 0·5 ml of methylene blue indicator solution and 10 ± 0·5 ml of chloroform. Titrate with the cationic reagent to the end point, rocking the titration vessel after each addition to avoid formation of emulsion and maintaining the temperature within 20°C and 30°C by immersion in water if necessary. After the first few additions, the blue colour concentrates entirely in the chloroform layer, but as the titration proceeds, there is a slow transfer of the colour to the water layer. As the end point is approached the rate of transfer of colour increases and the cationic reagent shall be added dropwise with vigourous shaking after each addition. If the approximate titration value is known, 80 percent of the required titrating solution should be added before shaking since this avoids emulsion trouble. Application of vacuum to the titration vessel may help in breaking an emulsion, if formed. The end point is reached when both the layers have same colour intensity. The end point is very sharp and 0·05 ml of the titrant will cause a distinct change in colour distribution at or near the equivalence point.

NOTE — The titration value should be as near to 10 as possible, values in the range 8 or 12 are acceptable but they shall never be less than 5 or more than 15.

A-2.1.3 *Calculation*

a) Calculate the combined sulphur trioxide content A from the following formula:

$$A\text{, percent by mass} = \frac{800{\cdot}0\,TN}{M}$$

where

T = volume in ml of standard cationic reagent used in the titration;

N = normality of standard cationic reagent [*see* **A-2.1.1.2** (b)]; and

M = mass in g of the material taken for the test in **A-2.1.2.2** (a).

b) From the combined sulphur trioxide content calculate sodium lauryl ether sulphate content as given below:

$$\text{Sodium lauryl ether sulphate, percent} = \frac{A\,(M' + 102)}{80}$$

where

M' = molecular mass of ethoxylated alcohol as declared by the manufacturer.

A-3. DETERMINATION OF NON-DETERGENT ORGANIC MATTER

A-3.0 Outline of the Method — From an aqueous solution of material unsulphated alcohols are extracted out with petroleum ether and then weighed after removal of solvent.

A-3.1 Reagents

A-3.1.1 *Potassium Hydroxide Solution* — Approximately 2 N.

A-3.1.2 *Rectified Spirit* — 95 percent (*v*/*v*), neutral to phenolphthalein.

A-3.1.3 *Phenolphthalein Indicator Solution* — Dissolve 0·1 g of phenolphthalein in 60 ml of rectified spirit and dilute with water to 100 ml.

A-3.1.4 *Petroleum Ether* — Boiling range 60 to 80°C.

A-3.1.5 *Alcohol Wash Solution* — 10 percent aqueous solution of rectified spirit.

A-3.2 Procedure

A-3.2.1 Weigh accurately about 20 g of the material. Dissolve in 50-ml of hot water and transfer to a 250-ml extraction cylinder using sufficient water to bring the volume to about 70 ml Add potassium hydroxide solution to make the solution neutral to phenolphthalein and then add 3 ml of the solution in excess. Add rectified spirit to bring the volume to about 150 ml. Cool well below the boiling point of petroleum ether.

A-3.2.2 Extract with petroleum ether at least 5 times using 50 ml portions of the solvent. In each extraction shake vigorously for 30 seconds. Allow to settle well between extractions. Addition of a few grams of sodium dihydrogen phosphate crystals eliminates formation of emulsion. Draw off petroleum ether layers into a small separating funnel and add 30 ml of alcohol wash solution and shake. Draw off and reject the water layer. Repeat washing until aqueous layer is neutral to phenolphthalein. If the washed petroleum ether extract is not clear, filter through a dry filter paper, washing it well with petroleum ether.

A-3.2.3 Remove the solvent from the clear petroleum ether extract by evaporation on a steam-bath out of direct contact with steam. A gentle stream of dry, clean, oil-free air or nitrogen shall be directed over the surface of the extract being evaporated. When the volume of the liquid gets reduced to about 25 ml, it shall be transferred to a weighed dish, washing twice with small portions of petroleum ether. The evaporation shall then be continued in the dish. Remove the dish containing the residue from the bath immediately after the last trace of the solvent has evaporated. Dry it in a desiccator and weigh.

A-3.3 Calculation

$$\text{Unsulphated fatty alcohols, percent by mass} = \frac{100\, M_1}{M_2}$$

where

M_1 = mass in g of the residue in the dish; and
M_2 = mass in g of the material taken for the test.

A-4. DETERMINATION OF SODIUM CHLORIDE

A-4.0 Outline of the Method — Chlorides are estimated by titration against silver nitrate solution.

A-4.1 Reagents

A-4.1.1 *Dilute Nitric Acid* — (1 : 20) (*v*/*v*).

A-4.1.2 *Potassium Chromate Indicator Solution* — 5 percent solution (*see* IS : 2263-1962*).

A-4.1.3 *Silver Nitrate Solution* — 0·1 N (*see* IS : 2316-1968†).

A-4.2 Procedure — Dissolve about 5 g of the material, accurately weighed, in about 50 ml of water, neutralize the solution with dilute nitric acid using litmus paper as indicator. Add a few drops of potassium chromate indicator solution and titrate with the silver nitrate solution till a permanent reddish tinge just appears.

A-4.3 Calculation

$$\text{Sodium chloride, percent by mass} = \frac{5{\cdot}845\, VN}{M}$$

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*Methods of preparation of indicator solution for volumetric analysis.

†Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

where

V = volume in ml of standard silver nitrate solution used in the titration;

N = normality of standard silver nitrate solution; and

M = mass in g of the material taken for test.

A-5. DETERMINATION OF SODIUM SULPHATE

A-5.0 Outline of the Method — Sodium sulphate is separated from sodium lauryl ether sulphate using rectified spirit. It is then determined by precipitation as barium sulphate.

A-5.1 Reagents

A-5.1.1 *Rectified Spirit* — 95 percent (*v*/*v*), neutral to phenolphthalein.

A-5.1.2 *Concentrated Hydrochloric Acid* — Conforming to IS : 265-1976*.

A-5.2 Procedure — Weigh accurately about 5 g of the material in a beaker. Heat with 200 ml of rectified spirit on a steam-bath for 2 hours. The mixture shall be alkaline to phenolphthalein throughout this heating period. Filter the solution and reject the filtrate. Transfer the residue with washing, to a beaker, add 200 ml of water and neutralize with concentrated hydrochloric acid adding about 0·5 ml of the acid in excess. If the solution is not clear, it shall be filtered and the filter paper washed until free from chlorides. The volume of the solution at this stage shall be 250 to 300 ml. Carry out the determination for sulphates as prescribed in IS : 2317-1975†.

A-5.3 Calculation

$$\text{Sodium sulphate (as } Na_2SO_4 \text{), percent} = \frac{60{\cdot}68\, M_1}{M_2}$$

where

M_1 = mass in g of the precipitate; and

M_2 = mass in g of the material taken for the test.

A-6. DETERMINATION OF CLOUD POINT

A-6.0 Principle — A stirred sample is cooled and the temperature at which solid matter other than ice separates is noted.

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*Specification for hydrochloric acid (*second revision*).

†Method for gravimetric determination of sulphates (*first revision*).

A-6.1 Apparatus — Test tube — 200 × 40 mm or 150 × 25 mm with ring type glass stirrer.

Thermometer — 10 to 50°C graduated in 0·1°C divisions.

Cooling Bath — A bath of a mixture of ethylene glycol and water fitted with a stirrer and cooled externally in freezing mixture.

A-6.2 Procedure

a) Place 25 ml of sample in the 40 mm tube (or 15 ml in the 25 mm tube);

b) Clamp the tube so that at least half of it is immersed in the cooling bath. Introduce thermometer and stirrer; and

c) Start cooling and stir the sample as it cools, taking care to scrape the sides of the tube and note temperature at which solid matter other than ice separates. This is the cloud point.

A-7. DETERMINATION OF VISCOSITY

A-7.0 Apparatus

a) Brookfield Synco Electric Viscometer model LV or RV;

b) Sample Vessel : Double walled glass or SS container of 100 ml capacity similar to a tall form beaker fitted with an inlet and outlet for water circulation; and

c) Constant temperature water bath which can be controlled to ±0·1°C.

A-7.1 Procedure — Transfer about 70 ml of the sodium lauryl ether sulphate solution to the sample vessel and start circulation of water from the constant temperature water bath. After the sample attains the temperature of 27 ± 0·1°C (it may be necessary to maintain the constant temperature bath at a slightly higher temperature say 27·5°C for the sample to reach 27°C) measure the viscosity using spindle No. 3 and an rpm value of 30.

A-8. DETERMINATION OF HEAVY METALS

A-8.0 Outline of the Method — The material is ashed and then extracted with a mineral acid. The colour produced by the solution with hydrogen sulphide solution is matched against that produced with standards.

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50-ml capacity (*see* IS : 4161-1967*).

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*Specification for Nessler cylinders.

A-8.2 Reagents

A-8.2.1 *Concentrated Hydrochloric Acid* — Conforming to IS : 265-1976*.

A-8.2.2 *Dilute Hydrochloric Acid* — 1 : 1 (*v*/*v*).

A-8.2.3 *Ammonium Chloride*

A-8.2.4 *Dilute Acetic Acid* — 1 N.

A-8.2.5 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate in water, add 1 ml of concentrated nitric acid (conforming to IS : 264-1976†) and make up the volume to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of the diluted solution contains 0·01 mg of lead (as Pb).

A-8.2.6 *Hydrogen Sulphide Solution* — Freshly prepared, saturated solution.

A-8.3 Procedure — Heat 5·000 g of the material in a silica crucible in a muffle furnace at 475 to 500°C. Cool and treat the residue with 3 drops of concentrated hydrochloric acid. Evaporate to dryness over a low flame and heat again in the muffle furnace for 20 to 30 minutes. A clean white ash should result, otherwise, the treatment may be repeated. Dissolve the white ash in a 1 ml of dilute hydrochloric acid and wash several portions of water into an evaporation dish. Evaporate to dryness on a steam-bath and dissolve the residue in about 30 ml of water. Transfer the solution to a Nessler cylinder, add 1 g of ammonium chloride and 1 ml of dilute acetic acid. Carry out a control test in another Nessler cylinder using 1·5 ml of standard lead solution in place of the material and 1 g of ammonium chloride and 1 ml of acetic acid. To each Nessler cylinder add 10 ml of hydrogen sulphide solution, dilute to the mark and shake well. Compare the intensity of colour produced in the two tubes.

A-8.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour obtained with material is not greater than that obtained in the control test.

A-9. TEST FOR ARSENIC

A-9.0 Outline of the Method — After incinerating the material, the ash is subjected to the Gutzeit test, the stain produced is compared with a standard stain.

A-9.1 Procedure

A-9.1.1 *Preparation of Solution* — Weigh 1·000 g of the material in a

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*Specification for hydrochloric acid (*second revision*).

†Specification for nitric acid (*second revision*).

platinum dish and incinerate for about 2 hours at 200 to 250°C. Cool and treat with a mixture of 5 ml of concentrated sulphuric acid and 5 ml of concentrated nitric acid. Take to fumes on a hot plate, cool and make up the volume to 100 ml.

A-9.1.2 Take 10 ml of the solution prepared in **A-9.1.1** and carry out the test for arsenic as prescribed in IS : 2088-1983*, using for comparison a stain obtained with 0 002 mg of arsenic trioxide.

A-10. DETERMINATION OF *p*H

A-10.1 Apparatus

A-10.1.1 *p*H *Meter* — Provided with glass and calomel electrodes.

A-10.2 Procedure — Heat 5 g of the sample in 100 ml of water to 80°C. Cool and filter. Determine the *p*H of the filtrate using the *p*H meter.

A-11. TEST FOR IRON

A-11.0 Outline of the Method — The colour produced with thiocyanate is matched against that obtained with standard iron solution.

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinders* — 50-ml capacity.

A-11.2 Reagents

A-11.2.1 *Concentrated Hydrochloric Acid* — *see* IS : 265-1976†.

A-11.2.2 *Ammonium Persulphate or Potassium Persulphate*

A-11.2.3 *Potassium Thiocyanate Solution* — 20 percent

A-11.2.4 *iso-Butanol*

A-11.2.5 *Standard Iron Solution* — Dissolve 0·700 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)2SO_4.7H_2O$] in 10 ml of dilute sulphuric acid (10 percent *v*/*v*) and dilute with water to 1 000 ml. Take 10 ml of the solution and dilute to 100 ml. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-11.3 Procedure — Dissolve 5·000 g of the material in 50 ml of water and 25 ml of concentrated hydrochloric acid. Add 0·5 g of ammonium or potassium persulphate, mix, heat to boil for about one minute and then cool. Make up the volume with water to 250 ml. Transfer 50 ml of the solution into a 100 ml separating funnel, and 10 ml of potassium thiocyanate solution and then 15 ml of *iso*-butanol. Stopper the funnel,

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*Methods for determination of arsenic (*second revision*).

†Specification for hydrochloric acid (*second revision*).

shake vigorously and allow the layers to separate. Transfer the aqueous layer to a second separating funnel and extract again with 15 ml of *iso*-butanol. Transfer the butanol, layer to the first separating funnel and extract the aqueous layer once again with 15 ml of *iso*-butanol. Combine all the butanol layers in a Nessler cylinder, make up the volume to 50 ml with *iso*-butanol and then mix. Carry out a control test using 2 ml of standard iron solution in place of the material and the same quantities of other reagents. Compare the colour produced in the two tubes after two minutes.

A-11.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-12. DETERMINATION OF FORMALDEHYDE

A-12.0 Principle — Formaldehyde reacts with acetyl acetone in the presence of ammonium acetate with the formation of a yellow lutidine derivative. This is extracted into *n*-butanol and the absorbance of the extract measured at 410 nm.

A-12.1 Reagents

A-12.1.1 *Glacial Acetic Acid — See* IS : 695 - 1975*.

A-12.1.2 *Anhydrous Ammonium Acetate*

A-12.1.3 *n-butanol*

A-12.1.4 *Acetyl Acetone Reagent* — Dissolve 150 g ammonium acetate, 2 ml acetyl acetone (fresh sample, if necessary, freshly distilled under reduced pressure but exhibiting no absorption at 410 nm) and 3 ml acetic acid in water and make up to 1 litre. Prepare immediately before use. (The *p*H should be about 6·4.)

A-12.1.5 *Formaldehyde Stock Solution* — Dilute 5 g of 37 to 40 percent formaldehyde solution to 1 litre. Check the concentration as follows:

> Pipette 10 ml, add 10 ml 0·1 N iodine solution and 10 ml N-sodium hydroxide solution. Stand for 5 minutes, then add 11 ml of N-hydrochloric acid and starch indicator solution. Titrate excess iodine with 0·1 N sodium thiosulphate solution. One ml 0·1 N iodine solution consumed = 1·5 mg formaldehyde.

A-12.1.6 *Formaldehyde Reference Solution* — Dilute 5 ml of stock solution to 100 ml with water. Dilute 5 ml of stock solution to 500 ml

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*Specification for acetic acid (*second revision*).

with water. 1 ml of this solution contains about 1 μg formaldehyde. Calculate the exact amount from the formaldehyde stock solution assay.

(CAUTION — POISON)

A-12.2 Procedure — Weigh a suitable quantity of sample to contain about 150 μg formaldehyde to an accuracy of 1 mg. Dissolve in water and dilute to 100 ml. Place 10 ml sample solution and 5 ml acetyl acetone reagent in a 50 ml Erlenmeyer flask. Dilute to about 30 ml with demineralised water and mix well. Immerse in a water bath at 60°C for exactly 10 minutes. Allow to cool for 2 minutes in an ice-bath. Transfer to a 50 ml separating funnel containing 10 ml *n*-butanol. Shake vigorously for exactly 30 seconds and allow the phases to separate. Run off the water layer and filter the butanol layer into a small stoppered flask through Whatman No. 1 or equivalent. This can be speeded by the using a phase separation filter, if available. Measure the absorbance at 410 nm in a 10 mm cell against a reference blank carried through the procedure.

NOTE — All operations must be completed within 25 min from the moment when the flask is placed in the bath at 60°C.

A-12.2.1 *Calibration Curve* — To prepare the calibration curve, place 5 ml of formaldehyde reference solution and 5 ml of acetyl acetone reagent in a 50 ml Erlenmeyer flask. Dilute to about 30 ml with demineralised water and mix well. Follow from "Immerse in water ...... a phase separation filter if available" as given in **12.2**, both inclusive, given under 'Analysis of Sample'. Measure absorbance at 410 nm in a 10 mm cell against *n*-butanol. Repeat the procedure as given under 'Calibration Curve' with 10, 15, 20 and 25 ml of formaldehyde reference solution.

A-12.3 Calculation — Plot the calibration curve — concentration as a function of absorbance. Beers Law is obeyed up to 30 μg of formaldehyde:

Mass (μg) of formaldehyde in the sample solution from the calibration curve = C

Mass of sample taken in g = W

$$\text{Formaldehyde percent} = \frac{C}{W \times 10^3}$$

APPENDIX B

(*Clause* 3.1)

SAMPLING OF SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with suitable stopper after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Sample shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers n to be chosen from the lot shall depend on the size of the lot N and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clause* B-2.2)

LOT SIZE (*N*)	NO. OF CONTAINERS TO BE SELECTED (*n*)
Up to 100	3
101 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7
1 001 and above	10

B-2.3 The containers to be selected for sampling shall be chosen at random. In order to ensure randomness of selection, random number tables shall be used for guidance and use of random number tables, IS : 4905-1968* may be referred. In the absence of random number tables, the following procedure may be adopted.

> 'Starting from any containers in the lot, count them as 1, 2, 3 . . . up to *r* and so on in one order, where *r* is the integral part of *N*/*n*, *N* being the lot size and *n* the sample size. Every *r*th container thus counted shall be withdrawn from the lot so as to constitute the required sample size'.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **3** and shall be not less than 300 gms.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal qnantity shall be taken from each selected container and shall be well mixed up together so as form a composite sample weighing not less than 0·5 kg. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to throughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

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*Methods for random sampling.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (**B-3.1.2**) and a set of individual samples (**B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of active detergent, percent by mass shall be conducted on each of the individual samples constituting the set of the test samples (*see* **B-3.1.3**).

B-4.2 Tests for the remaining characteristics, as given in **3** shall be conducted on the composite sample (*see* **B-3.1.2**).

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — The mean and range of test results for sodium lauryl ether sulphate shall be calculated as follows:

Mean ($\bar{X}$) = the sum of the test results divided by the number of test results; and

Range (R) = the difference between the maximum and the minimum values of the test results.

B-5.1.1 The lot shall be declared to have satisfied the requirement for sodium lauryl ether sulphate if $\bar{X} = 0{\cdot}6$ R is equal to or greater than the value specified in Table 1.

B-5.2 For Composite Sample — The test results on the composite sample shall meet the corresponding requirements given in Table 1 except active detergent, percent by mass which is tested on individual sample as in **B-5.1**.

B-5.3 A lot shall be declared as conforming to this specification if it satisfies the requirements for each of the characteristics prescribed. If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of the specification.

(*Continued from page* 2)

Members	*Representing*
SHRI V. R. RATHI	Sudershan Chemical Industries Ltd, Pune
SHRI U. N. LIMAYE (*Alternate*)	
DR S. K. ROY	Central Drugs Laboratory, Calcutta
DR A. C. DAS GUPTA (*Alternate*)	
DR M. P. SAHAKARI	Italab Pvt Ltd, Bombay
SHRI M. R. PAREKH (*Alternate* I)	
SHRI K. S. SANTHANAM (*Alternate* II)	
DR N. D. SHAH	Geoffrey Manners & Co Ltd, Bombay
DR M. M. DOSHI (*Alternate*)	
SHRI NAVINCHANDRA R. SHAH	Devarsons Pvt Ltd, Ahmadabad
SHRI VINODCHANDRA K. MEHTA (*Alternate*)	
SHRI M. S. THAKUR	Godrej Soaps Ltd,Bombay
SHRI N. G. IYER (*Alternate*)	

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s(s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

AMENDMENT NO. 1 MARCH 1999
TO
IS 11487 : 1985 SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

[*Page* 4, *Table* 1, *Sl No.* (x)] — Insert Sl No. (xi) and (xii) after (x):

(1)	(2)	(3)	(4)
xi)	Transmittance, percent, *Min*	85	A-13
xii)	Total microbial count, cfu/ml, *Max*	1 000	(IS 14648 : 1998)

(*Page* 15, *clause* A-12.3) — Insert a new clause 'A-13' after 'A-12.3':

A-13 DETERMINATION OF TRANSMITTANCE

A-13.1 Outline of the Method — The transmittance of 50 percent aqueous solution of sodium lauryl ether sulphate is spectrophotometrically determined in 1 cm fused silica cell at 420 nm.

A-13.2 Apparatus

A-13.2.1 *Spectrophotometer*

A-13.2.2 *Fused Silica Cells* — 2, 1 cm.

A-13.3 Procedure

A-13.3.1 *Spectrophotometric Measurements* — Weigh accurately 50.0 ± 0.1 g of the sample, dissolved in 50 ml of distilled water and mix well. Filter the solution through Whatman filter paper to remove any undissolved foreign material. Bring the solution to room temperature and let it stand until all the air bubbles disappear. Fill one of the fused silica cells with the test solution and fill the other cell with water. Carry out the spectrophotometric measurements at 420 nm after having adjusted the instrument to 100 transmittance against water.

A-13.3.2 *Correction for Transmittance of Cells* — Fill the two cells used for the measurements (A-13.3.1) with distilled water and measure the transmittance of each cell at a wavelength of 420 nm. Let one of the cells read '100' since it has been used to adjust the instrument to 100 transmittance.

A-13.4 Calculation — Calculate the transmittance of aqueous solution of SLES as under:

$$\begin{array}{l}\text{Transmittance of SLES (aqueous solution)}\\ \quad\text{in 1 cm fused silica cell at 420 nm}\end{array} = \frac{T_1 - T_0}{1}$$

where

T_1 = transmittance of test solution,

T_0 = correction for the differences in transmittance of the cells, and

l = optical path length of the cell, cm.

(PCD 19)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 DECEMBER 2002
TO
IS 11487 : 1985 SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

(*Page* 3, *clause* **2.1.1**, *line* 2) — Substitute 'liquid' *for* 'solution'.

[*Page* 4, *Table* 1, *Sl No.* (xii) (*see also Amendment No.* 1)] — Insert the following after Sl No. (xii):

(1)	(2)	(3)	(4)
xiii)	1,4 Dioxane, on 100% basis, ppm, *Max*	100	A-14

(*Page* 6, *clause* **A-2.1.2.1**, *line* 6) — Substitute'conical/iodine flask' *for* 'beaker'.

(*Amendment No.* 1, *clause* **A-13.4**) — Insert the following new clause after **A-13.4**:

A-14 DETERMINATION OF 1, 4 DIOXANE BY GAS CHROMATOGRAPHY METHOD UTILISING HEAD SPACE TECHNIQUE

A-14.1 Outline of the Method — Gas chromatographic method for the estimation of the presence of 1, 4 dioxane in sodium lauryl ether sulphate is given below both for packed column and capillary column. In case of dispute, Gas Chromatographic method using capillary column shall be considered as referee method.

A-14.2 Principle

The head space technique takes advantage of the volatility of 1,4 dioxane over the non-volatile ionic constituents of ethoxylated alcohol sulfate. Only the vapours above the liquid sample are injected onto the chromatographic column, preventing contamination of the column as compared to the direct injection techniques. Reproducibility is ensured by the use of an internal standard added to the sample with a retention time similar to 1,4 dioxane. The relative volatility of 1,4 dioxane is increased by adding N,N-dimethyl formamide to the sample matrix enhancing the sensitivity of the technique.

A-14.3 Gas Chromatographic Method Using Packed Column

A-14.3.1 *Apparatus*

a) Any gas chromatograph equipped with head space analyser, flame ionization detector (FID), and data handling package including CRT screen and printer.

The GC operating conditions are given below for guidance only:

Column	Packed		
Material	Stainless steel		
Length	6 feet		
I.D	1/8 inch		
Stationery Phase	Tenax >60/80 mesh		
Carrier gas	Helium, 30 ml/min		
Temperatures	Temp ($^{\circ}$C)	Time (min)	Rate ($^{\circ}$C/min)
Headspace Sampler	70	30	
First Oven Isothermal	170	6	
Oven Ramp Rate			30
Second Oven Isothermal	240	4	
Injector	290		
Flame Ionization Detector	290		

Others

FID Sensitivity — High

Timed Events for Headspace Sampler

Time (min)	Event	Position
-1.01	Hold	
-0.01	Relay 0	on
0.00	Relay 1	on
0.10	Relay 1	off
0.11	Relay 0	off

Data Acquisition

Start time	2.50 minutes
End Time	6.00 minutes
Width	-4
Detector Area Sens	25 to 500
Detector Base Sens	10 to 15
Calculation Type	Internal standard
1,4-Dioxane Retention Time (min)	3.1
Toluene Retention Time (min)	4.1

The calibration is accomplished by substituting 30% active ethoxylated alcohol sulfate with 30 percent active sodium lauryl sulfate and in place of the internal standard solution, a calibration solution is used. The details of calibration solutions are as follows:

Calibration solution — 50 mg/litre 1,4-dioxane + 50 mg/litre toluene in N,N-dimethylformamide.

Internal Standard — 50 mg/litre toluene in N,N-dimethylformamide

The standard analysis procedure is as follows:

b) Analytical balance — with accuracy of 0.000 1 g.

A-14.3.2 *Reagents*

A-14.3.2.1 *1,4-dioxane* — 99.9% purity.

A-14.3.2.2 *Toluene* — HPLC Grade

A-14.3.2.3 *N,N-dimethyl formamide* — 99.9% purity.

A-14.3.2.4 *Sodium lauryl sulfate, 30% active* — commercial material.

A-14.3.3 *Procedure*

0.5 g sample of 30 percent active ethoxylated alcohol sulfate is weighed into a vial with an accuracy of 0.1 mg. To the sample is added 1 ml of internal standard solution. The sample is sealed with a teflon faced septum using the crimping device. The sample vial is inserted into the heating block, equilibrated there for 30 minutes at 70°C. The chromatographic run is initiated manually by pushing a fixed injection needle through the septum of the vial, the valve relays direct the sample and helium flow. The result is that a portion of the vapour phase from the vial is injected onto the GC column. As the components of the sample vapour are resolved and exhausted to the FID the analog output is converted to digital output and collected by the data handling package. The toluene and 1,4 dioxane peaks are identified by retention times and the quantity of 1,4-dioxane is calculated from the relative peak areas of toluene and 1,4-dioxane, the result is output to the screen and printer along with a copy of the chromatogram. If any significant amount of a solvent such as ethanol is present in the ethoxylated alcohol sulfate sample then the calibration samples must include a like amount of the solvent. The chemical matrix of the sample effects the relative volatility of 1,4-dioxane and toluene and consequently the relative peak areas.

A-14.3.4 *Calculations*

Calibration factor F

$$F = \frac{\text{mg (1,4-dioxane)} \times \text{peak area (toluene)}}{\text{mg (toluene)} \times \text{peak area (1,4-dioxane)}}$$

1,4-dioxane in ppm (mg/kg)

$$\text{1,4-dioxane} = \frac{\text{mg (toluene)} \times \text{peak area (1,4-dioxane)} \times F \times 10^6}{\text{mg (sample weight)} \times \text{peak area (toluene)}}$$

A-14.4 Gas Chromatographic Method Using Capillary Column

A-14.4.1 *Outline of the Method*

The method describes the determination of 1,4-dioxane in alcohol ethoxysulphates up to 1 000 mg/kg.

A weighed amount of sample is analyzed after stabilization in a heated compartment using a Headspace sampler, by means of gas chromatography over a fused silica capillary column using a sample splitter and flame ionization detection. This method describes the determination of 1,4-dioxane in alcohol ethoxysulphates up to 1000 mg/kg.

A-14.4.2 *Apparatus*

A-14.4.2.1 Any Gas Chromatograph provided with a flame ionization detector, a sample splitter, temperature-programming facilities and a Headspace sampler.

Operating Instructions for Gas Chromatograph

Column	
Type	Capillary
Material	Fused Silica
Length, I.D.	60 m × 0.32 mm
Stationary Phase	Methyl silicone fluid
Film thickness	1 micron
Detector	
Type	Flame Ionization
Sample Splitter	Split ratio 1:30
Temperatures	
Column Oven	Initial temperature: 80°C hold for 7 minutes
	Final temperature: 250°C hold for 20 minutes
	Programming rate: 15°C/min
Injector	250°C

Head space sampler	Aux. temperature : 80°C
Detector	300°C
FID sensitivity	High
FID zero	On
Carrier Gas	Helium 1 ml/min
Pressure	120 kPa

Time in Headspace : 15 minutes

Sampler

Time events

Time :	1.01 min.	Event :	hold	
Time :	1.00 min.	Event :	relay 0 :	on
Time :	0.00 min.	Event :	relay 1 :	on
Time :	0.10 min.	Event :	relay 1 :	off
Time:	0.11 min.	Event :	relay 0 :	off

A-14.4.2.2 Injection vials 10 ml, with matching serum caps for safe closing.

A-14.4.3 *Reagents and Materials*

A-14.4.3.1 *1,4-Dioxane*, A.R.

Caution: 1,4-Dioxane presents a health hazard if incorrectly handled. Prevent contact with the skin. Avoid inhalation.

A-14.4.4 *Procedure*

A-14.4.4.1 Into two separate vials, accurately weigh 2 g of homogeneous sample, to the nearest 10 mg. Introduce an amount 1,4-dioxane to one of the vials to obtain an accurately known concentration of added 1,4-dioxane of approx. 100 mg/kg, and mix thoroughly. Seal both vials immediately in such way that no leakage can occur.

NOTE — Dilute weighed samples with water, if necessary and apply correction factor in calculations.

A-14.4.4.2 Place the vials subsequently into the head space sampler and warm for 15 minutes at 80°C and inject into the gas chromatograph according to the operating instructions given.

A-14.4.4.3 Determine the active matter content of the sample in % (m/m) as per procedure given in IS 4956 : 2002 'Synthetic detergents for industrial purposes (*second revision*)'.

A-14.4.5 *Interpretation of the Chromatogram*

The peak for 1,4 chromatogram appears at about 3 to 3.5. A typical chromatogram under the conditions given above is shown in Fig. 1.

A-14.4.6 *Calculation*

Calculate the 1,4-dioxane content of the sample by means of the following equation:

$$\text{1,4-dioxane, mg/kg} = \frac{A}{\frac{B\text{-}A}{D}} \times \frac{100}{C} \times K$$

where

A = peak area of 1,4-dioxane in sample without addition, arbitrary units;

B = peak area of 1,4-dioxane in sample after addition, arbitrary units;

C = active matter content of sample, percent (m/m);

D = concentration of 1,4-dioxane added to sample, mg/kg; and

K = sample dilution factor (if applicable).

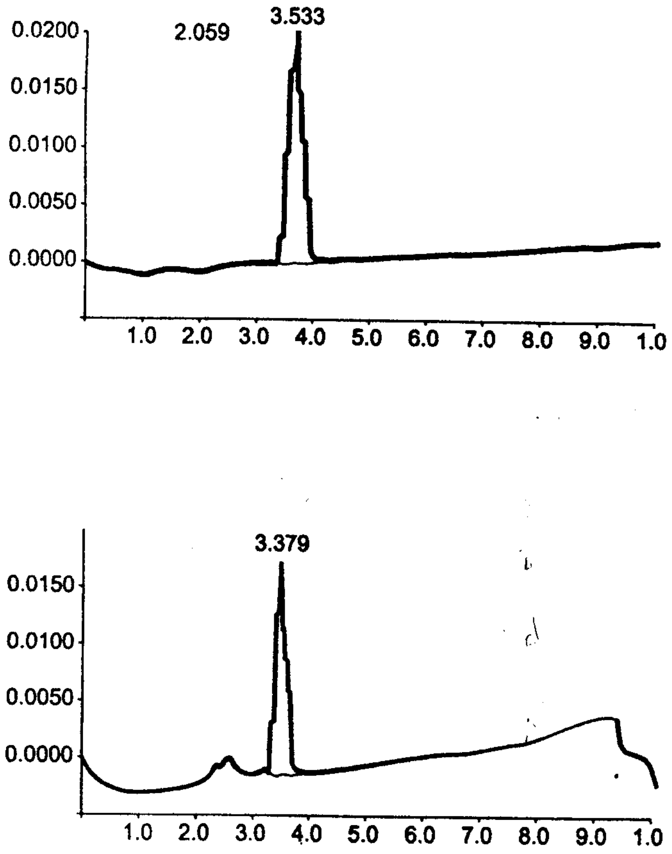

FIG. 1 A TYPICAL CHROMATOGRAM FOR 1, 4 DIOXANE

(PCD 19)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 SEPTEMBER 2012
TO
IS 11487 : 1985 SPECIFICATION FOR SODIUM LAURYL ETHER SULPHATE FOR COSMETIC INDUSTRY

[*Page* 4, *Table* 1, *Sl No.* (v)] — Delete.

[*Page* 4, *Table* 1, *Sl No.* (viii), *col* 3] — Substitute '6.0 to 11.0' *for* '6.0 to 8.0'.

[*Page* 4, *Table* 1, *Sl No.* (xiii), *col* 3, *see also Amendment No*. 2] — Substitute '30' *for* '100'.

(PCD 19)

Reprography Unit, BIS, New Delhi, India